

Chapter 18: Molecular Spectroscopy
A Non-Trivial Beer's Law Problem Involving Standard Addition

Nitrate ion in water is determined by reacting with phenoldisulfonic acid (PA), forming a yellow solution with $\lambda_{\text{max}} = 410 \text{ nm}$. A 100.0-mL sample with an unknown concentration of NO_3^- (FM 62.00) is treated with silver sulfate to precipitate chloride ion, which interferes with the phenoldisulfonic acid reaction. The precipitate is filtered and washed (with washings added to the filtered sample). The sample solution is adjusted to pH 7 with dilute NaOH and evaporated just to dryness. The residue is treated with 2.0 mL phenoldisulfonic acid solution and heated in a hot water bath to aid dissolution. Twenty mL distilled water and 6 mL ammonia are added to intensify the color of the sample, and the solution is transferred to a 50-mL volumetric flask and diluted to the mark with additional distilled water.

A blank is prepared using the same volume of reagents, starting with the disulfonic acid step. A standard nitrate solution is prepared by dissolving 0.722 g anhydrous KNO_3 (FM 101.1) and diluting to exactly 1 L. A standard addition calibration is performed by spiking a separate 100.0-mL portion of sample with 1.00 mL of the standard solution and carrying through the entire procedure. The following absorbance readings were obtained at 410 nm, using a cuvet 1.00-cm wide: blank, 0.032; sample, 0.270; sample plus standard, 0.854.

What is the concentration of nitrate in the sample in parts per million (ppm)?